



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: **Brian Gibson, et al.** Examiner: **William K. Cheung**

Application No.: **10/766,430**

Group Art: **1713**

Filing Date: **January 28, 2004**

Attorney Docket No.: **H0003690**

For: **EXTRUDABLE PVC COMPOSITIONS**

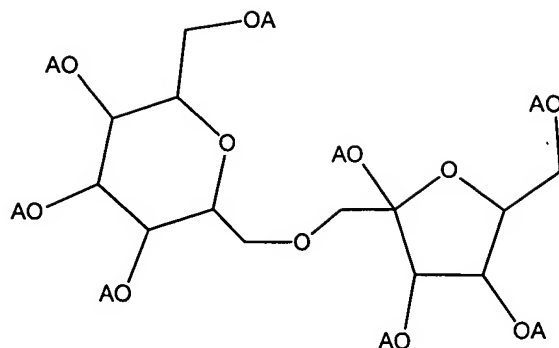
Commissioner for Patents  
Alexandria, VA 22313-1450

**RULE 132 DECLARATION OF FRED DURRENBERGER**

I, Fred Durrenberger, declare and state that:

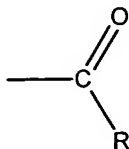
1. I am an inventor in the above-referenced patent application. I hold a B.S. degree in Chemical Engineering (1978) from University of Missouri-Rolla College. I have been employed by Honeywell International, Inc. and/or its predecessor in interest, the assignee of the application, since about 1992 holding various positions, including my current position as Director of Technology in the Wax and Additives Group. I have extensive experience and familiarity with the development of the extrusion methods and compositions which are the subject of the claims pending in this application.
2. I am familiar with the prosecution history of the above-mentioned patent application and understand that the claims under consideration in this application (as amended as of the date of this filing) are directed generally to a process for extruding a resin-containing composition comprising providing an extrudable mass containing at least one extrudable resin and saccharide ester comprising one or more compounds of in accordance with Formula I as specified in the claims. More particularly, the compounds of Formula I are specified in the claims as follows:

Formula I:



wherein "A" is hydrogen or has the structure of Structure I:

Structure I



wherein "R" is an aliphatic or aromatic moiety of about eight to about 40 carbon atoms, and wherein all of the "A" moieties of at least about 50 wt. % of the compounds of Formula I comprise moieties of Structure I.

3. I am aware that the Patent Office has rejected claims 1 – 18 and 67 of the present application (as previously presented) as being anticipated by US 3,635,856 (Kaneko). I have reviewed both the Office Action and the Kaneko patent and understand the contents of both.
4. I understand the Kaneko patent to relate to non-toxic compositions comprising PVC and stabilizer composed of several additives, including sucrose alkyl ester and polyhydric alcohols. This patent indicates that the purpose of including the sucrose in the composition is because of its interaction with the polyhydric alcohol, namely the formation of eutectic mixtures therewith and also to make certain metal salts included in the composition dispersible in and over the entire body of the PVC composition during the course of mixing. (see col. 3, lines 17 – 30). I also note that this patent teaches that the sucrose alkyl esters may be those having a hydrophile-lipophile balance (HLB) of below 6. However, this patent does not describe the manner by which this balance is calculated and it is difficult in the absence of such a description to assign meaning to this teaching. On the other hand, the patent seems to indicate that the sucrose alkyl esters are preferably diesters or monoesters of fatty acids having an alkyl group with 12 to 18 carbon atoms and mixtures thereof. This description is readily understood to mean that

the esters have either one or two of the hydroxyl groups on the sugar substituted by the ester. I note that, under certain techniques used today to measure what is referred to as HLB balance, such mono- and di-ester sugars would typically have HLB values of greater than 6 (see the chart attached as Exhibit A). Moreover, this patent further indicates that esters resulting from the complete esterification of organic acid have proven to be ineffective to prevent the shaped body to color in the early stages after molding. (col. 3, line 73 – col. 4, line 1). Based on this passage, and other passages (including those mentioned above), I believe that a person skilled in the art at the time our invention was made would believe that highly substituted esters would not have a heat stabilizing effect on PVC compositions. More specifically, I believe that the Kaneko patent, taken as a whole, would teach away from highly substituted esters, included particularly octa-substituted sucrose esters, and instead teaches that mono- and di- substituted sucrose esters should be used.

5. I have tested and/or supervised the testing of three different compositions. The first composition (Sample 1A) is made in accordance with the methods as now claimed in the present application, while the other two compositions (Samples 1B and 1C) are outside the scope of the claims as now amended. The results of these tests are reported in Table 1 below. Unless otherwise indicated, the materials used to prepare the various compositions were as follows: 1091® polyvinyl chloride resin (resin) from Georgia Gulf; TM281® tin based heat stabilizer (stabilizer) from Rohm & Haas; Rheolub® 165 paraffin wax (paraffin wax) from Honeywell, COAD 10® calcium stearate (calcium lubricant) from Norac; A-C® 629 oxidized polyethylene wax from Honeywell (polymer wax); Paraloid K120N® acrylic processing aid (processing aid) from Rohm & Haas; Paraloid KM334® acrylic impact modifier (impact modifier) from Rohm & Haas; UFT® calcium carbonate from OMYA (calcium carbonate); titanium dioxide (TiO<sub>2</sub>), and sucrose stearate obtained from Procter & Gamble. All materials were used as received.
6. The compositions in Table 1 were prepared by charging the constituent components into a Waring Blender (high intensity mixer) and heating to a temperature in excess of 105°C while operating the mixing blades. When the components were substantially homogeneously blended, the composition was discharged, cooled to about ambient temperature and left to stand for about 24 hours (aging period). At the end of the aging period a sample of about 68g of the composition was placed into a Brabender torque rheometer and subjected to rheometric analysis. Each composition was subjected to rheometric analysis of the fusion and stability time, equilibrium and fusion torque and equilibrium temperature substantially according to ASTM standard testing procedure D2538. The results are presented in Table 1 below.

Table 1

Parameter	Test Sample A	Test Sample B	Test Sample C
Degree of Substitution	At least 70 wt.% Octa	Estimated 40% mono- and 58% tri-(based on Chart attached as Exhibit 1)	Estimated 73% mono- and 27% tri-(based on Chart attached as Exhibit 1)
Equilibrium Temp (°C)	212	215	216
Fusion Torque (mg)	3,370	3,900	4,050
Equilibrium Torque (mg)	1,860	1930	1930
Fusion Time (min)	0.78	0.45	0.43
Dynamic Stability time, min.*	14	10	11

\*elapsed time between fusion and measurable onset of cross-linking (minutes)

7. As can be seen from Table 1, each of the three PVC resin-containing compositions were prepared substantially the same except that the specific saccharide ester composition used in each was different. More specifically, Test Sample 1A is based on a saccharide ester purchased from Proctor & Gamble under the trade designation Sefose-1618H. A copy of the MSDS sheet for this product is attached as Exhibit B. Test Sample 1B is based on a saccharide ester purchased from Mitsubishi-Kagaku Foods Corporation under the trade designation S-770. A copy of a data sheet for this product is attached as Exhibit C. Test Sample 1C is based on a saccharide ester purchased from Mitsubishi-Kagaku Foods Corporation under the trade designation S-1670. A copy of a data sheet for this product is attached as Exhibit D. The Sefose-1618H used for Sample 1A comprised at least about 70% by weight of octa-substituted sucrose stearate and had a hydrophilic/lipophilic balance (HLB value) calculated by modern methods of less than 1. The S-770 used for Sample 1B is believed to comprise about 40% by weight of mono-substituted sucrose esters, about 58% by weight of di-substituted sucrose esters, and to have a hydrophilic/lipophilic balance (HLB value) calculated by modern methods of about 7. The S-1670 used for Sample 1C is believed to comprise about 73% by weight of mono-substituted sucrose esters, about 27% by weight of di-substituted sucrose esters, and to have a hydrophilic/lipophilic balance (HLB value) calculated by modern methods of about 16. Each composition was subjected to the same Brabender torque rheometer

test and analysis as described above to determine fusion and stability time, equilibrium and fusion torque and equilibrium temperature substantially according to ASTM standard testing procedure D2538.

8. Table 1 illustrates applicants' discovery that the degree of substitution of the saccharide ester in accordance with the present invention has a significant beneficial effect on the fusion characteristics of the shapeable composition, particularly the equilibrium temperature, the fusion and equilibrium torque, fusion time and dynamic stability, with the higher level of substitution exhibiting superior performance than compositions based on lower levels of substitution. This table evidences that compositions comprising saccharide esters having a higher degree of substitution, particularly predominantly octa stearate as in the case of Test Sample A, exhibit properties characteristic of a higher degree of external lubrication than the lower substituted saccharide esters, as evidenced by the increased fusion time and reduced fusion torque. Furthermore, at the equilibrium condition, the melt temperature of the predominately octa substituted saccharide ester is considerably less than that of the lower substituted compounds, and the equilibrium torque is equal to or lower than that of the lower substituted compounds. Finally the dynamic heat stability appears to be beneficially impacted by the use of compositions based on a high degree of substitution; particularly compounds having predominantly octa substituted saccharides. The Kaneko patent does not in any way suggest this latter finding and in fact would lead a person skilled in the art away from this discovery.
9. I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true and further that the statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Fred Durrenberger

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Date

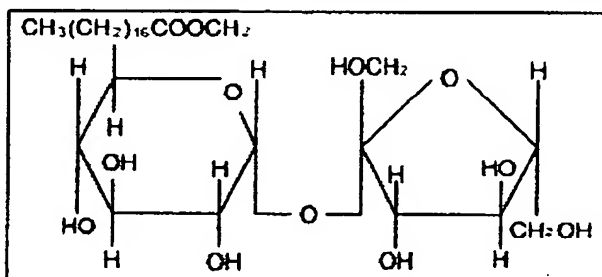
## Introduction of SUGAR ESTERS

### Chemical Structure of Sugar Esters

Sucrose fatty acid esters are nonionic surfactants consisting of sucrose as hydrophilic group and fatty acid as lipophilic group, and generally called Sugar Ester.

As sucrose has a total of 8 hydroxyl groups, compounds ranging from sucrose mono to octa fatty acid esters can be produced.

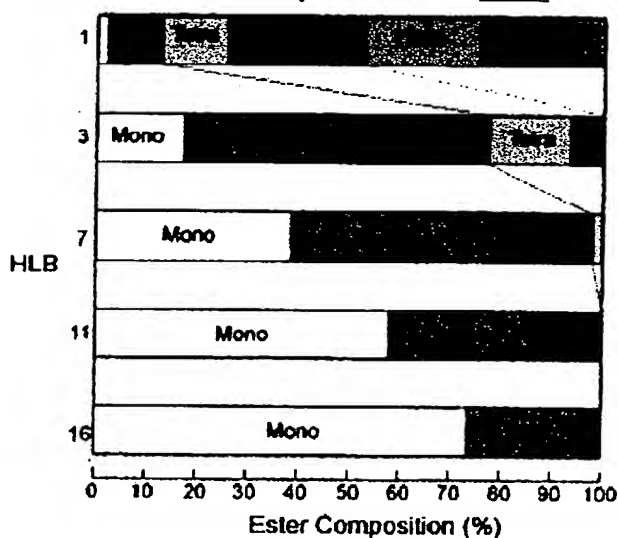
### Chemical Structure of Sucrose Monostearate

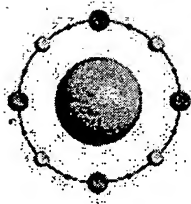


### Features of Sugar Esters

- Being tasteless, odorless and nontoxic, they are the best suited emulsifier for foods.
- Being non-irritant to the eyes and skin, they are suitable not only for foods but also for pharmaceuticals and cosmetics.
- Because of their excellent biodegradability they don't cause environmental pollution.
- Sugar Esters offer a full range of HLB values from 1 to 16, and in use all grades display exceptionally good surfactant functionality.

### Ester Composition and HLB





# Procter & Gamble Chemicals

Page 1 of 6

## MATERIAL SAFETY DATA SHEET

MSDS Number: SEFA503-2

Revision Date: September 20, 2002

Supersedes: SEFA503-1

Issue Date: August 20, 2002

### 1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND OF THE COMPANY/UNDERTAKING

- **Product identification**

Synonyms/Trade Names: SEFA Stearate, Hardened SEFA SOYATE / SEFOSE-1618H

- **Product uses**

Lubricant

- **Company/undertaking identification**

North America: The Procter & Gamble Company  
Procter & Gamble Chemicals  
Sharon Woods Technical Center  
11530 Reed Hartman Highway  
Cincinnati, Ohio 45241

Department issuing MSDS: Product Safety and Regulatory Affairs  
1-800-477-8899

Europe: Procter & Gamble European Supply Company N.V.  
P&G Chemicals – Europe  
The Heights  
Brooklands  
Weybridge  
Surrey  
ENGLAND KT13 0XP  
Telephone Number: 01932-896000

- **Emergency Telephone:**

P&G Ltd. - Brooklands, England:

CHEMTREC

Tel 01932-896000 (day phone)  
Emergency 0191-279-2000 (day phone)  
1-800-424-9300 U.S. and Canada  
1-703-527-3887 For calls originating  
elsewhere

U.S. Emergency, Quality or Service Issues:

Call Customer Service  
1-800-477-8899 or 513-626-6882

**2. COMPOSITION/INFORMATION ON INGREDIENTS**

Substance/Preparation (mixture): Substance

	<u>CAS No.</u>	<u>Wt/Wt %</u>	<u>EC-No.</u>	<u>EC Symbols</u>	<u>EC R-phrases</u>
alpha.-D-Glucopyranoside, .beta.-D-fructofuranosyl, octadecanoate	37318-31-3	100	2534596	Not applicable	Not applicable

Hazardous Ingredients as defined by OSHA 29 CFR 1910.1200: None

Occupational exposure limits, if applicable, are listed in Section 8

LC/LD50 information is listed in Section 11.

Full text of R phrase(s) are listed in section 16.

**3. HAZARDS IDENTIFICATION**

European Hazard Classification: This product is not classified as dangerous according to Directive 67/548/EC.

- Potential Health Effects:

Eye: Acute and chronic exposure - No effects reported in humans.

Skin: Acute and chronic exposure - No effects reported in humans.

Inhalation: Acute exposure - No demonstrated health effects. Worst-case (aspiration) may cause lipoid pneumonitis.

Chronic exposure- No demonstrated health effects. Repeated or prolonged exposure to very high levels may cause lipoid pneumonitis. Persons with a history of chronic respiratory disease may be at increased risk from severe exposure.

Ingestion - Acute exposure- Large quantities may have a laxative effect.

Chronic exposure- No known adverse health effects.

- Physical/Chemical Hazards: Not identified.

- Environmental Hazards: Not identified.

**4. FIRST AID MEASURES**

- Eye - Wash eyes immediately with large amounts of water until no evidence of material remains.
- Skin - Wash thoroughly with plenty of water and soap.
- Inhalation/aspiration - If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen. Keep warm and get medical attention.
- Ingestion - No action required. Induction of vomiting is not recommended.



**5. FIRE FIGHTING MEASURES**

- Extinguishing media: Dry chemicals, carbon dioxide, foam, or water spray.
- Unsuitable extinguishing media: Not available
- Flash Point and method: 530° F (Closed cup)
- Explosive limits in air:
  - Upper: Not applicable
  - Lower: Not applicable
- Auto-ignition temperature: Not available
- Smoke point: 450° F
- Sensitivity to mechanical impact/static discharge: Not available
- Special Protective Equipment: Wear self-contained breathing apparatus and full protective clothing.
- Other Fire Fighting Considerations: Cool containers with flooding quantities of water until well after fire is out.
- Exposure hazards: Not available

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**6. ACCIDENTAL RELEASE MEASURES**

- Personal Precautions: Wear an appropriate NIOSH/MSHA approved respirator, suitable gloves and eye/face protection.
- Environmental Precautions: Minimize contamination of drains, surface and ground waters.
- Procedures for Spill/Leak Clean-up: Handle as any grease or oily material.

Refer to Section 8 for additional personal protection information.

Refer to Section 13 for disposal considerations.

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**7. HANDLING AND STORAGE**

- Handling: Handle in accordance with good hygiene and safety procedures. Avoid contact with eyes, skin, and clothing. Wash thoroughly after handling. May be slippery.  
Since empty containers contain product residue, follow all hazard warnings and precautions even after container is emptied. Keep away from sources of ignition.
- Storage: Store in original closed containers in a cool, dry, well-ventilated area. Store separately from all combustible, organic and oxidizable materials. Store all materials in properly labeled containers.
- Specific use(s): Follow bulk handling and storage procedures as noted above.

Refer to Section 6 for clean-up of spillages.

Refer to Section 13 for disposal considerations.

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**8. EXPOSURE CONTROLS/PERSONAL PROTECTION**

- General Precautions: Good industrial hygiene should be followed.  
Avoid breathing (heated) vapors. Avoid eye and skin contact.
- Exposure Limit Values:  
ACGIH - Threshold limit value:  $10 \text{ mg/m}^3$ ; based on the TLV for similar lipid materials (i.e. vegetable oil mists).
- Exposure Controls:  
  
Engineering Controls: Ventilation: Provide local exhaust ventilation or general dilution ventilation to meet permissible exposure limits.  
  
Personal Protective Equipment  
  
Eye - None required; however goggles are recommended if working within splash distance of heated material.  
  
Skin - Protective gloves: Not required but recommended.  
Protective clothing: Not required but recommended if working within splash distance of heated material.  
  
Inhalation - An appropriate NIOSH/MSHA approved respirator should be used if a mist or vapor is generated.  
Respiratory protection must be used in accordance with the OSHA respirator standard (29 CFR 1910.134).
- Other Controls: None required.
- Environmental Exposure Controls: Contact Procter and Gamble for specific Community information.

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**9. PHYSICAL AND CHEMICAL PROPERTIES**

- General Information:  
  
Physical State @ 72° F: Waxy Solid  
Appearance: White  
Similar to edible fats and oils.  
Odor: Bland  
Odor Threshold: Not available
- Important health, safety and environmental information:  
  
pH: Not available  
Boiling point/Boiling range: Not applicable (will smoke, vaporize, and decompose rather than boil)  
Flash Point & Method: 530° F (Closed cup)  
Flammability (solid, gas): Not available  
Explosive properties: Not available  
Oxidising properties: Not available  
Vapor pressure: Not applicable (NA); material is inert  
Relative density: 0.90 to 0.95 at 160° F (fully melted)  
Melting point range: 135° F to 145° F (varies with fatty acid composition)

**9. PHYSICAL AND CHEMICAL PROPERTIES - CONTINUED**

## Solubility:

Water solubility: Insoluble

Fat solubility (solvent-oil to be specified): Not available

Partition coefficient: n-octanol/water: Not available

Viscosity, cps @ 121° C 20

177° C 7

Vapor Density (AIR=1): Not applicable

Evaporation Rate (nBuOAc=1): Not applicable (doesn't evaporate)

Explosive Limits: Not applicable

Auto ignition temperature: Not available

Coefficient of water/oil distribution: Not available

**10. STABILITY AND REACTIVITY**

- Stability: Stable under normal operational conditions.
- Conditions to Avoid: Oxygen at high temperatures (>120° F).
- Materials to Avoid: Avoid strong acids, strong bases, oxidizing agents
- Hazardous Decomposition Products: Not available
- Hazardous Polymerization: Not expected.

**11. TOXICOLOGICAL INFORMATION**

Available upon request.

**12. ECOLOGICAL INFORMATION**

Available upon request.

**13. DISPOSAL CONSIDERATIONS**

DISPOSAL IS TO BE PERFORMED IN COMPLIANCE WITH ALL FEDERAL, STATE/PROVINCIAL AND LOCAL REGULATIONS. Do not dispose of via sinks, drains or into the immediate environment.

**14. TRANSPORT INFORMATION**

Not regulated for transport.

**15. ADDITIONAL REGULATORY INFORMATION**

INVENTORY STATUS: TSCA (USA), NDSL (Canada), China, EINECS (EU), Korea, PICCS (Philippines)

**EC LABELING AND CLASSIFICATION:**

This product is not classified as dangerous according to Directive 67/548/EEC.

**15. ADDITIONAL REGULATORY INFORMATION - CONTINUED**

**Canada**

**HAZARDOUS INGREDIENTS – WHMIS (Canadian Workplace Hazardous Materials Information System)**

This product when tested as a whole is not a controlled substance within the meaning of the Hazardous Products Act.

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all the information required by the Controlled Products Regulations.

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**16. OTHER INFORMATION**

**EUROPE**

This product safety data sheet was prepared in compliance with 2001/58/EC.

The following section(s) contain revisions or new statements: 9

Department issuing MSDS: Product Safety and Regulatory Affairs 1-800-477-8899

The submission of the MSDS may be required by law, but this is not an assertion that the substance is hazardous when used in accordance with proper safety practices and normal handling procedures. Data supplied are for use only in connection with occupational safety and health.

The information contained herein has been compiled from sources considered by Procter & Gamble to be dependable and is accurate to the best of the Company's knowledge. The information relates to the specific product designated herein, and does not relate to use in combination with any other material of any other process. Procter & Gamble assumes no responsibility for injury to the recipient or third persons, or for any damage to any property resulting from misuse of the controlled product.



**MITSUBISHI-KAGAKU FOODS CORPORATION**  
 JITSUGYO NO NIHONSHA BLDG. 3-9, GINZA 1-CHOME, CHUO-KU, TOKYO 104-0061 JAPAN  
 PHONE : +81-3-3563-1510 FACSIMILE : +81-3-3563-1520

Date: November 19, 2004

To: Mitsubishi Chemical America, Inc.

HLB = 7

**CERTIFICATE OF ANALYSIS of Ryoto Sugar Ester S-770 for Food**

Name	Sucrose fatty acid esters
Code of Federal Regulations	Sucrose fatty acid esters is listed in the 21CFR section 172.859
Product Name (Used on Label)	Ryoto Sugar Ester S-770
Lot number	47078101
Manufacturing date	July 7, 2004
Best before date	July 6, 2006

	Items	Specifications	Results
1	Assay (Mono-, di-, and tri-esters)	Not less than 80.0 %	pass
2	Free sucrose	Not more than 4.0 %*	0.2%
3	Acid value	Not more than 5.0*	1.6
4	Residue on Ignition	Not more than 1.5 %*	1.0%
5	Arsenic (as As <sub>2</sub> O <sub>3</sub> )***	Not more than 1 mg/kg*	pass
6	Heavy metals (as Pb)	Not more than 10 mg/kg	pass
7	Lead***	Not more than 10 mg/kg	pass
8	Methyl ethyl ketone**	Not more than 10 mg/kg	pass
9	Ethyl acetate**	Not more than 350 mg/kg	pass
10	Methanol	Not more than 10 mg/kg	pass
11	Dimethylsulfoxide	Not more than 2 mg/kg	pass
12	Isobutanol	Not more than 10 mg/kg	pass
13	Moisture***	Not more than 4.0 %	0.3%

\* : Lower than that defined in Food Chemicals Codex (1996)  
 \*\* : Not used in the manufacturing process  
 \*\*\* : Not defined in Food Chemicals Codex (1996)

MITSUBISHI-KAGAKU FOODS CORPORATION

*Mitsumasa Kaitou*  
 Mitsumasa Kaitou  
 General Manager  
 Quality Assurance Department



# MITSUBISHI-KAGAKU FOODS CORPORATION

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 PHONE : +81-3-3563-1510 FACSIMILE : +81-3-3563-1520

Date: April 15, 2005

To: Mitsubishi Chemical America, Inc

HLB = 16

## CERTIFICATE OF ANALYSIS of Ryoto Sugar Ester S-1670 for Food

Name	Sucrose fatty acid esters
Code of Federal Regulations	Sucrose fatty acid esters is listed in the 21CFR section 172.859
Product Name (Used on Label)	Ryoto Sugar Ester S-1670
Lot number	53098101
Manufacturing date	March 9, 2005
Best before date	March 8, 2007

	Items	Specifications	Results
1	Assay (Mono-, di-, and tri-esters)	Not less than 80.0 %	pass
2	Free sucrose	Not more than 4.0 %*	0.1%
3	Acid value	Not more than 5.0*	0.9
4	Residue on ignition	Not more than 1.5 %*	0.2%
5	Arsenic (as As <sub>2</sub> O <sub>3</sub> )***	Not more than 1 mg/kg*	pass
6	Heavy metals (as Pb)	Not more than 10 mg/kg	pass
7	Lead***	Not more than 10 mg/kg	pass
8	Methyl ethyl ketone**	Not more than 10 mg/kg	pass
9	Ethyl acetate**	Not more than 350 mg/kg	pass
10	Methanol	Not more than 10 mg/kg	pass
11	Dimethylsulfoxide	Not more than 2 mg/kg	pass
12	Isobutanol	Not more than 10 mg/kg	pass
13	Moisture***	Not more than 4.0 %	0.4%

\* : Lower than that defined in Food Chemicals Codex (1996)  
 \*\* : Not used in the manufacturing process  
 \*\*\* : Not defined in Food Chemicals Codex (1996)

MITSUBISHI-KAGAKU FOODS CORPORATION

*Mitsumasa Kaitoh*  
 Mitsumasa Kaitou  
 General Manager  
 Quality Assurance Department